

STANDARD OPERATING PROCEDURE FOR FILTERING WATER COLUMN CHLOROPHYLL-A SAMPLES

State of Utah
Department of Environmental Quality
Division of Water Quality



Revision 1
Effective May 21, 2013

Utah Division of Water Quality (DWQ) Standard Operating Procedures (SOPs) are adapted from published methods, or developed by in-house technical experts. This document is intended primarily for internal DWQ use. This SOP should not replace any official published methods.

Any reference within this document to specific equipment, manufacturers, or supplies is only for descriptive purposes and does not constitute an endorsement of a particular product or service by the author or by DWQ. Additionally, any distribution of this SOP does not constitute an endorsement of a particular procedure or method.

Although DWQ will follow this SOP in most instances, there may be instances in which DWQ will use an alternative methodology, procedure, or process.¹

¹ *Disclaimer language above adapted from Washington State Department of Ecology SOPs.*

REVISION PAGE

| Date | Revision # | Summary of Changes | Sections | Other Comments |
|---------|------------|---|----------|--|
| 5/21/13 | 1 | Draft SOP was updated to include new laboratory method (corrected for pheophytin) and sample label. | 8 & 10 | New SOP. Began document control/revision tracking. |

TABLE OF CONTENTS

| | | |
|------|---|----|
| 1.0 | Scope and Applicability | 5 |
| 2.0 | Summary of Method | 5 |
| 3.0 | Definitions and Acronyms | 5 |
| 4.0 | Health and Safety Warnings | 6 |
| 5.0 | Cautions | 6 |
| 6.0 | Interferences..... | 6 |
| 7.0 | Personnel Qualifications/Responsibilities | 7 |
| 8.0 | Equipment and Supplies..... | 7 |
| 9.0 | Procedure | 8 |
| 10.0 | Laboratory Analysis | 10 |
| 11.0 | Data and Records Management..... | 11 |
| 12.0 | Quality Assurance and Quality Control | 11 |
| 13.0 | References | 12 |
| 14.0 | Appendix | 13 |

1.0 SCOPE AND APPLICABILITY

This document presents the Utah Division of Water Quality's (DWQ) Standard Operating Procedure (SOP) for field processing of water column samples from Utah's wetlands, lakes, and rivers/streams for chlorophyll-a analysis (phytoplankton method). This SOP does not cover benthic chlorophyll-a sample collection for the UCASE Program (Utah Comprehensive Assessment of Stream Ecosystems). The UCASE chlorophyll-a method (periphyton method) is covered in the UCASE Field Operations Manual.

This SOP gives procedures for the field filtering of water samples for chlorophyll-a; it does not discuss collection of water samples. Water sample collection should be performed in accordance with specific program SOPs or field manuals, or a specific project sampling and analysis plan (SAP). This SOP applies to all personnel collecting chlorophyll-a samples including DWQ monitors, non-DWQ State of Utah cooperators, and volunteer monitors.

Chlorophyll-a is a green photosynthetic pigment and concentrations of chlorophyll-a are used to infer algal biomass. The DWQ uses these data in several ways. As an important indicator for lake water quality assessment, chlorophyll-a concentrations are one component used to calculate the Carlson Trophic State Index, a measure of the degree of eutrophication in a lake/reservoir. Chlorophyll-a concentrations in rivers/streams are used to support nutrient criteria development. Chlorophyll-a is also included in a suite of water chemistry parameters evaluated as a component of a Multimetric Index (MMI) used to assess wetland condition.

Note: This field filtering method may also be used to process water column AFDW (ash-free dry weight or ash-free dry mass) samples.

2.0 SUMMARY OF METHOD

A filter apparatus comprised of a hand pump or Geopump™ (peristaltic pump), plastic tubing, a filter flask, and filter funnel with a filter stage is used to filter the water sample. An unused glass-fiber filter is placed on the filter stage. Sample water is poured into the filter funnel and then pumped through the filter, at a vacuum pressure not to exceed 7 psi. A volume of 500 ml is typically filtered; however, turbid water may clog the filter, resulting in a lower sample volume. The filter (containing the residue to be analyzed) is removed from the filter stage and wrapped in foil or placed into an opaque sample container. The total volume filtered is recorded and the sample is frozen until extracted and analyzed by the laboratory.

3.0 DEFINITIONS AND ACRONYMS

DI: deionized water

| | |
|----------|---|
| ml: | milliliter(s) |
| mm: | millimeter(s) |
| psi: | pounds per square inch |
| residue: | The part of the sample remaining as a solid on the filter paper after the liquid passes through the filter. |
| RPM: | revolutions per minute |
| µm: | micrometer(s), also called microns |

4.0 HEALTH AND SAFETY WARNINGS

Not applicable to this SOP. See SOPs for sample collection.

5.0 CAUTIONS

IMPORTANT: If a sample is turbid, filter only small volumes of the sample at a time. A turbid sample will not filter to 500 ml. Keep track of the volume filtered and record the final volume on the lab sheet and field sheet/notes. Submit only one (1) filter to the laboratory, regardless of the final volume, unless otherwise directed by the project manager or project-specific SAP.

When using a hand pump or Geopump™, be careful to not exceed 7 psi (14 inches of Hg) when applying vacuum pressure. High pressures can rupture algal cell walls allowing the chlorophyll-a pigment to pass through the filter and not be included in the analyzed sample, leading to underestimated concentrations or false negative results.

6.0 INTERFERENCES

A dirty filter apparatus can lead to sample contamination. The apparatus and graduated cylinder should be cleaned prior to a sampling trip by washing or overnight soaking in Liquinox followed by a light scrubbing. After cleaning, the stage and flask should be thoroughly rinsed with tap water followed by a thorough rinse with DI water and then air dried. The apparatus should be decontaminated between samples during a trip by rinsing the filter stage and filter flask 3 times with deionized (DI) water and shaking dry. The stage and flask should be inspected for any adhered particles. If noticeable buildup is on the stage or flask they should be soaked in soapy water and then scrubbed until buildup is removed.

Highly turbid water may cause the filters to clog more frequently. If sample water is turbid, filter only a small volume at a time.

Chlorophyll-a pigment is broken down by exposure to sunlight (photodecomposition). Therefore the filtering procedure should be carried out in subdued lighting conditions, if feasible. The filter apparatus should be set up prior to collection of the sample, the sample bottle should be protected from sunlight once collected, and the sample should be filtered as soon after sampling as possible. In addition, processed and frozen filters should also be protected from light during storage by using an appropriate opaque sample container such as aluminum foil and placed in a zip top bag to prevent contamination. Prepared samples must be stored in a cooler with dry ice or placed in a freezer immediately to prevent sample degradation.

7.0 PERSONNEL QUALIFICATIONS/RESPONSIBILITIES

Samplers are required to read this SOP annually and acknowledge they have done so via a signature page that will be kept on-file at DWQ along with the official hard copy of this SOP (see **Appendix**). Personnel collecting chlorophyll-a samples must be trained by an experienced DWQ employee. Demonstration of capability may be required for cooperators and volunteer monitors on a yearly basis. Yearly review sessions will be held by the State of Utah to review procedures and should be attended by all personnel intending on sampling for chlorophyll-a.

8.0 EQUIPMENT AND SUPPLIES

- _____ Copy of this SOP
- _____ Field notebook, lab sheet
- _____ Pens, pencils
- _____ Sample labels (**Figure 1**)
- _____ Sample containers for filters (aluminum foil and zip top bag is preferable, another clean opaque container is an acceptable alternative)
- _____ Polypropylene graduated cylinder, 500 ml
- _____ Glass fiber pre-filters (Grade GF/F, 47 mm diameter, 0.7 μ m pore size)
- _____ Plastic disposable filter forceps, length: 4 1/2" (2 pair aids in filter folding)
- _____ DI water in squeeze bottle
- _____ Cooler and dry ice (if unable to put samples in freezer immediately after processing)
- _____ Hand pump (25" Hg, 15cc pumping rate/stroke) and Tygon or similar tubing (approximately 24"), diameter needs to fit filter flask arm, **OR**
- _____ Geopump™ with quick-release pump head, fitted with approximately 3 feet of Masterflex tubing attached, and an inline pressure gauge on the vacuum side of the tubing
- _____ Nalgene or similar polypropylene filter flask, 500 ml
- _____ Nalgene analytical test filter funnels
- _____ #7 filter flask stoppers with 1/2 -inch hole

Figure 1. Chlorophyll-a sample label.

(U:\WQ\PERMITS\MONITORS\Labels\Chlorophyll-a(5162)_010113.doc)

| | |
|--|-----------------|
| <u>Chlorophyll-a by SM10200H(2)(b) (Dry-Ice)</u> | |
| SITE ID: _____ | |
| _____ | |
| STORET: _____ | |
| DATE: _____ | SAMPLERS: _____ |
| FIELD METHOD (Circle One): WATER / ALGAE / NDS | |

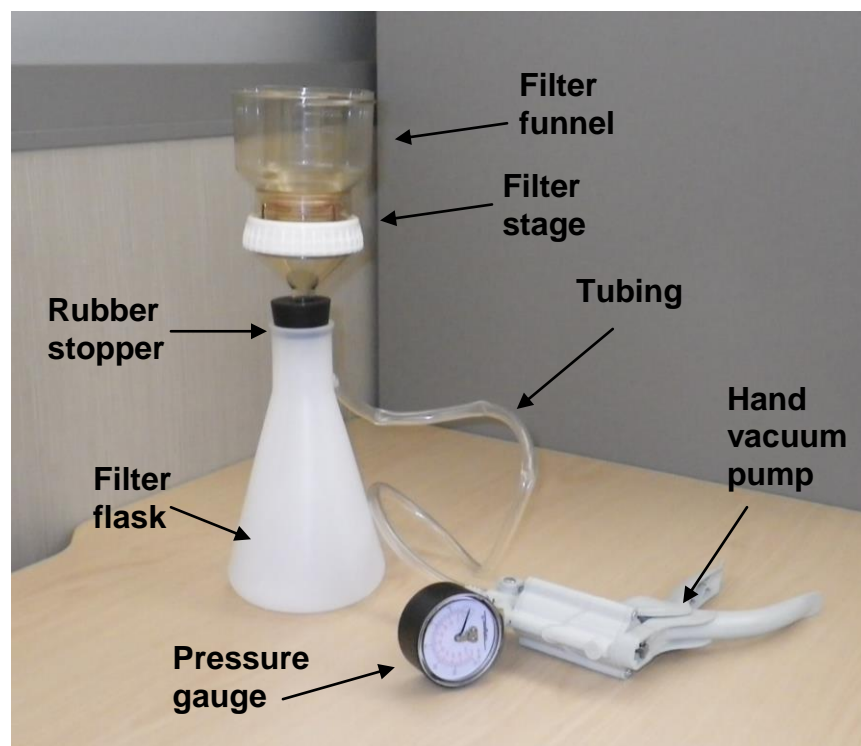
9.0 PROCEDURE

This procedure can be carried out using a Hand Pump or a peristaltic pump (such as a Geopump) outfitted with a vacuum pressure gauge. Regardless of what pumping method is used, the same filter flask and filter holder can be used, and operating pressures must not exceed 7 psi.

Start each sampling trip with equipment that has been cleaned with soap (liquinox) and water, rinsed 3 times with tap water, rinsed 3 times with DI water, and allowed to dry.

- 1) Attach tubing onto filter flask arm and pump.
- 2) Insert the funnel adapter into the #7 stopper; wetting the adapter may aid in this. Once the adapter is in the stopper it need not be disassembled unless a thorough cleaning of the entire apparatus is required.
- 3) Insert the filter funnel assembly into the top of the filter flask until it seals tightly. **Figure 2** provides a photograph of the entire filter apparatus assembly attached to a hand-pump.
- 4) Remove the top of the filter funnel from the filter stage.
- 5) Using the forceps, place an unused glass fiber filter on the filter stage.
- 6) Wet the sides of the filter funnel and filter with DI water from the squeeze bottle to create a good seal on the filter.
- 7) Mix the water sample in its container multiple times to homogenize, and pour the sample into a graduated cylinder to measure 500 ml (or a smaller volume if sample is turbid).
- 8) Pour 250 ml (or a smaller volume) of sample water into the filter funnel and use the pump to pull the sample through the filter.

Figure 2. Filter apparatus with a hand pump.



- 9) Continue filtering measured volumes of sample, being careful not to exceed 7 psi of vacuum pressure during filtration. When up to 500 ml of sample water has been filtered, or the filtration rate has slowed, or the filter has turned a green/brown color, sufficient sample has been filtered.

NOTE: *If the sample is turbid, only filter small volumes of sample at a time. Try to estimate how much turbid water you can put through a filter by paying attention to how the pressure increases and the flow-rate decreases as you pull the water through the filter. If you overestimate the volume of water that can pass through the filter and water is left remaining on top of a clogged filter, filtering must be repeated with another volume of sample and a new filter.*

- 10) Rinse down the sides of the filter funnel with DI water and use the pump to pull the rinse water through the filter. You can stop filtering when the filter is still slightly wet; the filter need not be completely dry.
- 11) Remove the filter funnel being careful not to disturb the filter stage or filter.
- 12) Using forceps, remove the filter from the filter stage, being careful not to rip the filter or scrape off any green/brown residue.
- 13) Using forceps, fold the filter in half with the residue facing the inside.

- 14) Place the folded filter onto a piece of aluminum foil and fold the foil to make a package for the filter. Alternatively, place the folded filter into a clean opaque sample container (such as a clean plastic film canister).
- 15) If using foil, place the foil-wrapped sample into a small ziptop bag.
- 16) Fill out the sample label and drop into or adhere onto the ziptop bag containing the sample. If using an opaque container, attach the label to the container using clear tape.
- 17) Place the sample into a larger ziptop bag and store the sample immediately on dry ice or place in the freezer.
- 18) Deliver the sample(s) to the laboratory as soon as possible. Samples must be analyzed within 3 weeks (21 days) of collection.

Decontamination Procedure: *Between samples, triple-rinse the graduated cylinder, filter funnel, filter flask, and forceps with DI water and shake dry as best as possible.*

10.0 LABORATORY ANALYSIS

Filters are extracted and analyzed for chlorophyll-a by Standard Method 10200 H.2.b (homogenization followed by spectrophotometry). The methodology and quality assurance and quality control procedures for this analysis and analyzing laboratories can be obtained from:

Unified State Laboratories: Public Health, Utah Department of Health
4431 South 2700 West
Taylorsville, UT 84119
(801) 965-2400
UPHL@utah.gov

NOTE: *The sample processing procedure described in this SOP can also be used to prepare filters for ash free dry weight (AFDW) analysis. Filters are analyzed by the State Laboratory using Standard Methods (American Public Health Association, 1999, section 10300 C.6). See **Figure 3** for AFDW sample labels.*

Figure 3. AFDW sample label.

(U:\WQ\PERMITS\MONITORS\Labels\AFDW(5162)_010113.doc)

| | |
|--|---------------------|
| <u>AFDW by SM10300C-Periphyton (Dry-Ice)</u> | |
| SITE ID: | _____ |
| _____ | |
| STORET: | _____ |
| DATE: | _____ |
| SAMPLERS: | _____ |
| FIELD METHOD (Circle One): | WATER / ALGAE / NDS |

11.0 DATA AND RECORDS MANAGEMENT

The volume of sample water filtered must be recorded in two places: on the lab sheet and in the field notes. In addition, the field method ("WATER" for water column samples) *must be circled on the sample label (Figure 1)*. Once the laboratory has received the samples, the lab sheets are photocopied and copies sent back to DWQ. In addition, the volume of water filtered should be recorded in the electronic field data file for the sampling trip. See DWQ's Field Data Management SOP for detailed instructions.

12.0 QUALITY ASSURANCE AND QUALITY CONTROL

Field quality control samples to be performed with chlorophyll-a sampling include blanks and duplicates.

For blanks, decontaminate the filtering apparatus as described in **Section 9.0**. Then filter 500 ml of DI water through the filter apparatus and process as a regular sample. At a minimum one blank should be performed per sampling trip or per every 10 samples to check for cross-contamination between samples. Some other frequency may be required according to a program or project-specific quality assurance plan or SAP.

For the field duplicate, filter a second volume of water from the same sampling container. It is very important to completely homogenize the water sample before pouring into the filter funnel both for the regular and the duplicate sample. Filter and process the field duplicate as a regular sample. The filtering apparatus should be decontaminated as described in **Section 9.0** between processing the regular and duplicate sample. At least one field duplicate should be performed for every 20 regular samples, or at a frequency indicated in a program or project-specific SAP.

13.0 REFERENCES

Hambrook Berkman, J.A., and Canova, M.G., 2007, Algal biomass indicators (ver. 1.0): U.S. Geological Survey Techniques of Water-Resources investigations, book 9, chap. A7, section 7.4, August, accessed__date__ from <http://pubs.water.usgs.gov/twri9A/>.

Lazorchak, James M., Donald J. Klemm, and David V. Peck. 1998. Environmental Monitoring and Assessment Program – Surface Waters: Field Operations and Methods For Measuring the Ecological Condition Of Wadeable Streams. Washington D.C.: U.S. Environmental Protection Agency.

Standard Methods for the Examination of Water and Wastewater, 20th Edition, 1998, Method 10200H “Chlorophyll”.

USEPA. 2010. Sampling and analytical procedures for GLNPO’s Open Lake Water Quality Survey of the Great Lakes. EPA 905-R-05-001. Great Lakes National Program Office, U.S. Environmental Protection Agency, Chicago, Illinois. (<http://www.epa.gov/glnpo/monitoring/sop/index.html>)

Related DWQ SOPs:

Utah Comprehensive Assessment of Stream Ecosystems (UCASE) Field Operations Manual

Standard Operating Procedure for Collection of Lake Water Samples

Standard Operating Procedure for Collection of Water Chemistry Samples

14.0 APPENDIX

SOP Acknowledgement and Training Form

This SOP must be read and this form signed annually. This form must be kept with the current version of the SOP.

| | |
|---------------------------|--|
| Document Title: | |
| Document Revision Number: | |
| Document Revision Date: | |

Please sign below in accordance with the following statement: “I have read and understood the above referenced document. I agree to perform the procedures described in this SOP in accordance with the document until such time that it is superseded by a more recent approved revision.”

| Printed Name | Signature | Date |
|--------------|-----------|------|
| | | |
| | | |
| | | |
| | | |
| | | |
| | | |
| | | |
| | | |
| | | |
| | | |
| | | |
| | | |
| | | |
| | | |
| | | |
| | | |
| | | |
| | | |
| | | |

